



Chemically extracted nanocellulose from sisal fibres by a simple and industrially relevant process

Trifol Guzman, Jon; Sillard, Cecile; Plackett, D.; Szabo, Peter; Bras, Julien; Daugaard, Anders Egede

Published in:
Cellulose

Link to article, DOI:
[10.1007/s10570-016-1097-5](https://doi.org/10.1007/s10570-016-1097-5)

Publication date:
2017

Document Version
Peer reviewed version

[Link back to DTU Orbit](#)

Citation (APA):
Trifol Guzman, J., Sillard, C., Plackett, D., Szabo, P., Bras, J., & Daugaard, A. E. (2017). Chemically extracted nanocellulose from sisal fibres by a simple and industrially relevant process. *Cellulose*, 24(1), 107–118.
<https://doi.org/10.1007/s10570-016-1097-5>

General rights

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

Supplementary Information

Chemically extracted nanocellulose from sisal fibers by a simple and industrially relevant process

J. Trifol^a, C. Sillard^c, D. Plackett^b, P. Szabo^a, J. Bras^c, A. E. Dugaard^a

^a Danish Polymer Centre, Department of Chemical and Biochemical Engineering, Technical University of Denmark, Søtofts Plads, Building 229, DK – 2800 Kgs. Lyngby, Denmark

^b Faculty of Pharmaceutical Sciences, University of British Columbia, 2405 Wesbrook Mall, Vancouver, BC V6T 1Z3, Canada

^c LGP2/Grenoble INP-Pagora/CNRS, 461 rue de la papeterie, Domaine universitaire, C10065, 38402 Saint Martin d'Hères Cedex, France

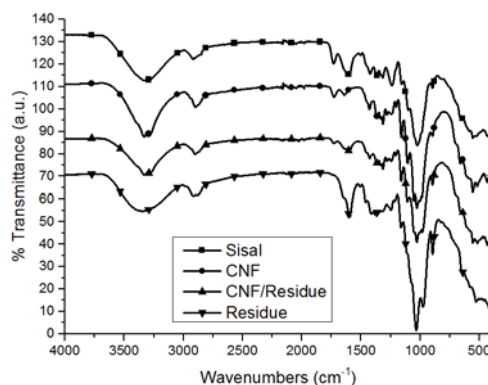


Figure 1 FTIR of Sisal, CNF, CNF/Residue and residue

The residue was characterized by FTIR (SI-Figure 1). As can be the residue shows a peak at 3330 cm⁻¹ which is characteristic of the cellulose and a very clear peak at 1590 cm⁻¹ which is characteristic of the lignin, meaning that we have both lignin and cellulose present at the residue. Although the peak at 1740 cm⁻¹, characteristic of the hemicellulose, has disappeared from the spectra of the residue, does not mean that there is no hemicellulose present, the reason is that as has been reported (Patil et al., 2012) the strong alkali treatments can deacetylate the hemicellulose. This is also supported by the fact that this peak it is neither present in SM (fig 4). Finally, it has been reported already that the alkali treatment extracts hemicellulose from fibers (Fang et al., 2000) so it can be concluded that there is cellulose, hemicellulose and lignin present in the residue.

It can also be seen how the FTIR spectra of the sisal, the CNF and CNF/residue can be compared in order to see if the CNF/residue had a similar composition as the sisal and as can be seen on fig 4, there are only minor

differences between both spectra. The peak at 1594 cm^{-1} (lignin) is slightly smaller in the CNF/residue than in the sisal, but it is slightly higher than in the CNF. The reason is that the alkali treatments can not remove all of the lignin (for that reason there is a mass loss in the following procedures and the fiber after the alkali treatment is still brown), meaning that with the current procedure we can recover part of the lignin but not all of it.

The behavior of the peak at 1740 cm^{-1} is more complex. This peak is indicative of the acetyl groups of the hemicellulose, but also of the grafted acetyl groups on the CNF. As long as the residue is rich on hemicelluloses, it was expected to have a more intense signal on this band higher on the CNF/residue than on the CNF, but in fact it is lower.

This is probably due to the fact that the alkali treatments deacetylate the hemicelluloses (Patil et al., 2012), moreover, as long as in the CNF/residue the concentration of CNF is smaller, the signal of the acetate groups of the CNF should be smaller, as it is. Finally this conclusion is supported by the fact that the peak at 3330 cm^{-1} (attributed to the OH stretching of cellulose molecules) is more intense in CNF than in CNF/residue, meaning that the CNF has higher cellulose content than CNF/residue.

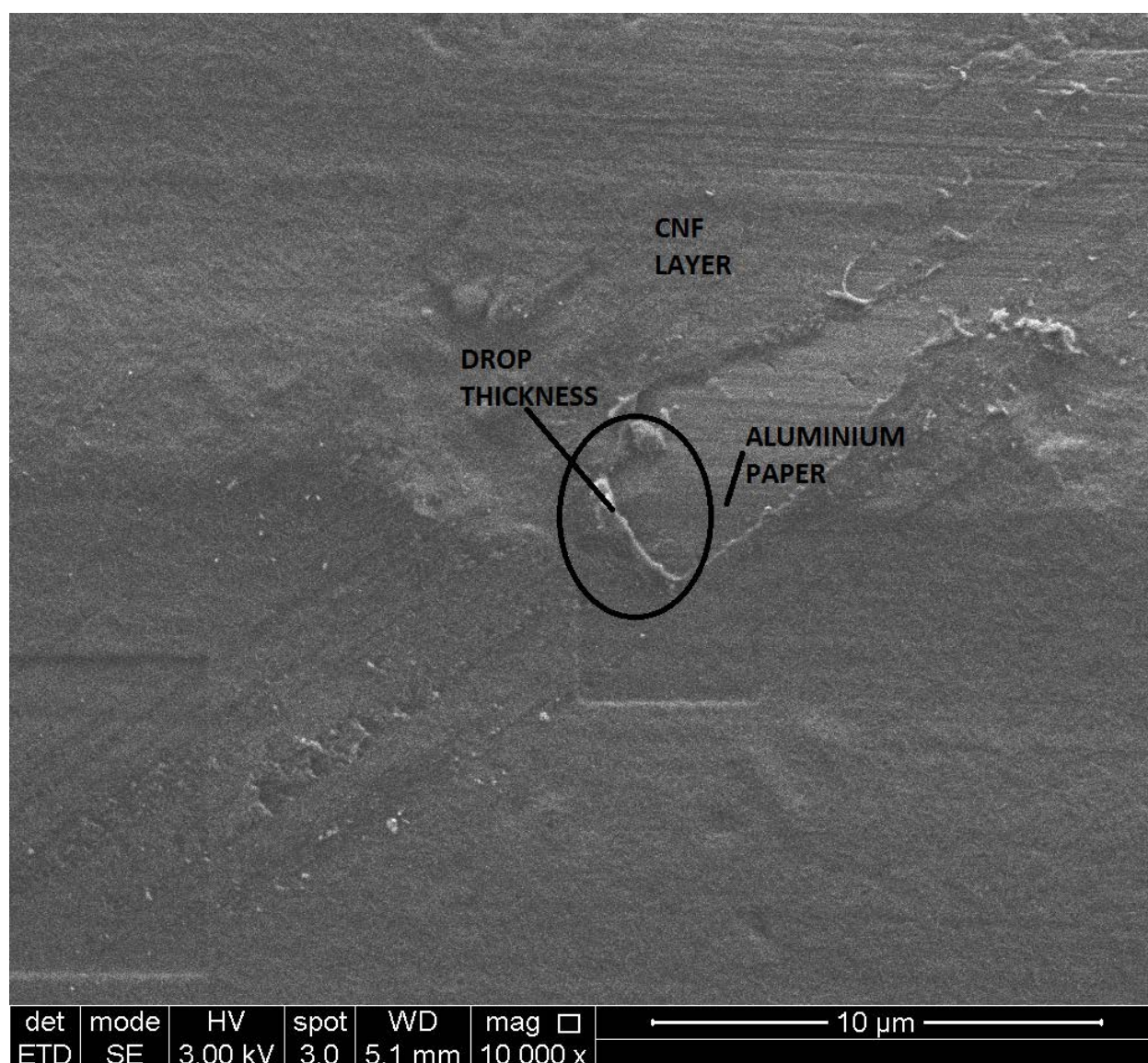


Figure 2a SEM micrographs of a casted drop in an aluminum foil.

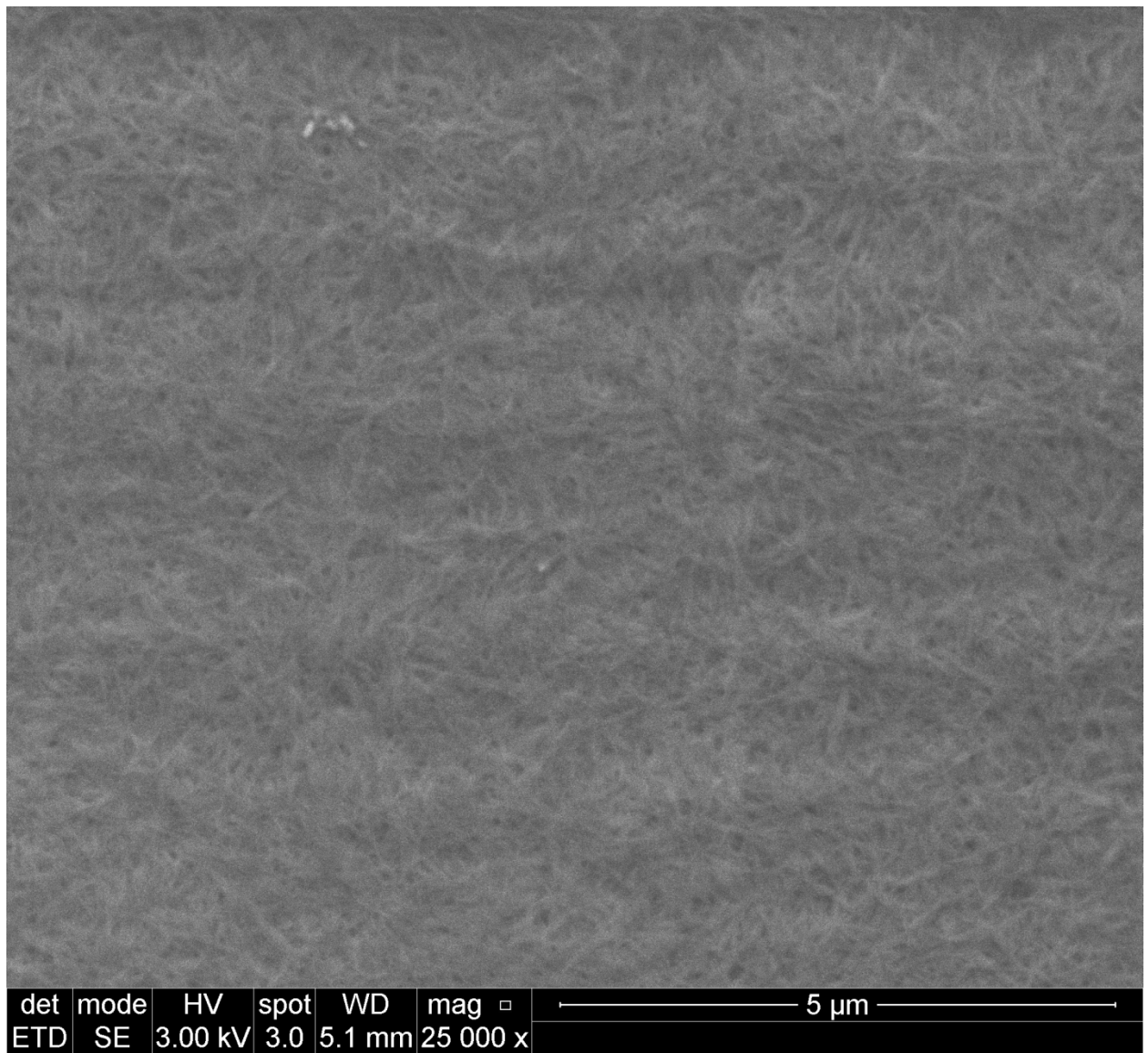


Figure 2b SEM micrographs of a casted drop in an aluminum foil.

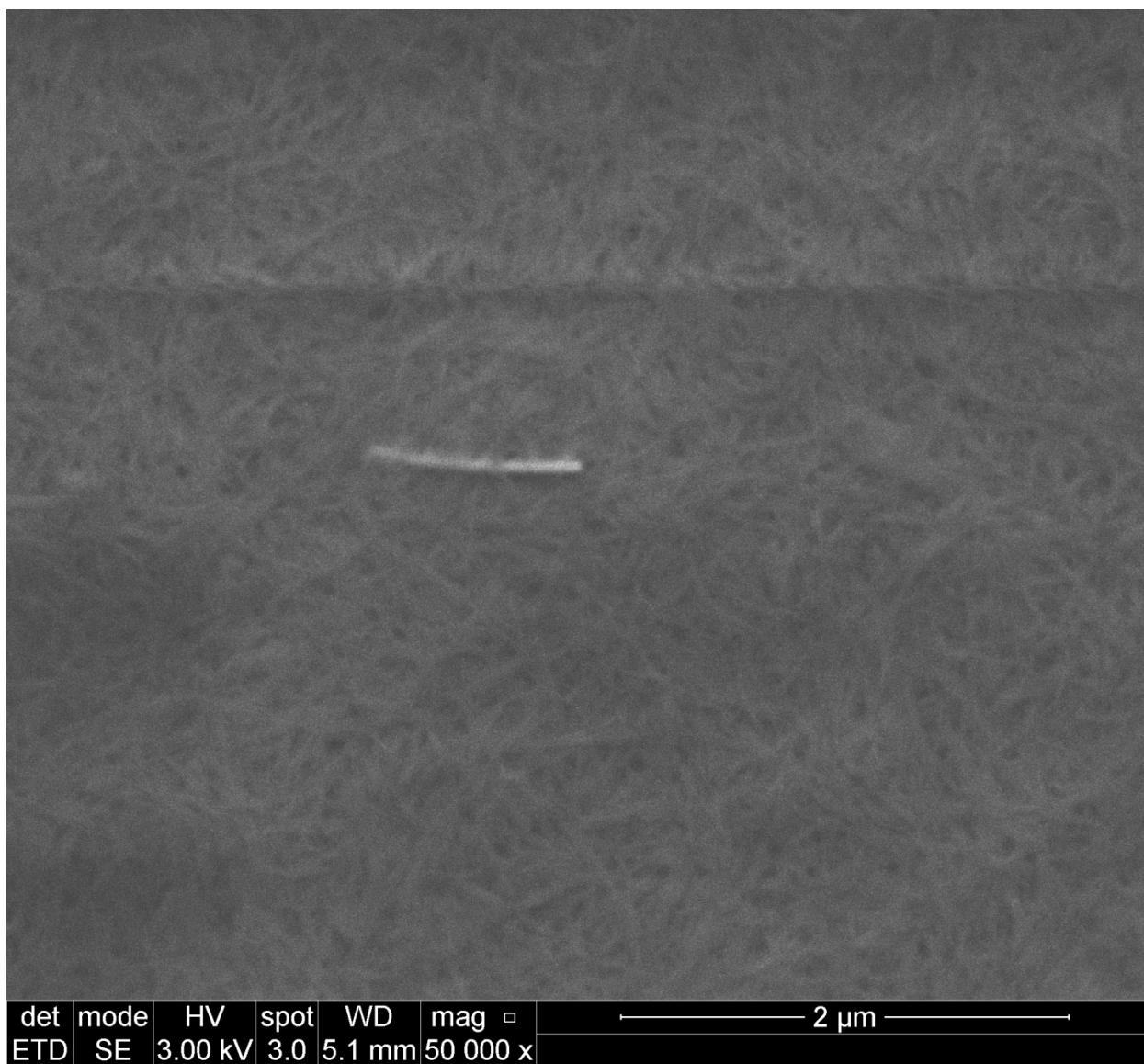


Figure 2c SEM micrographs of a casted drop in an aluminum foil.

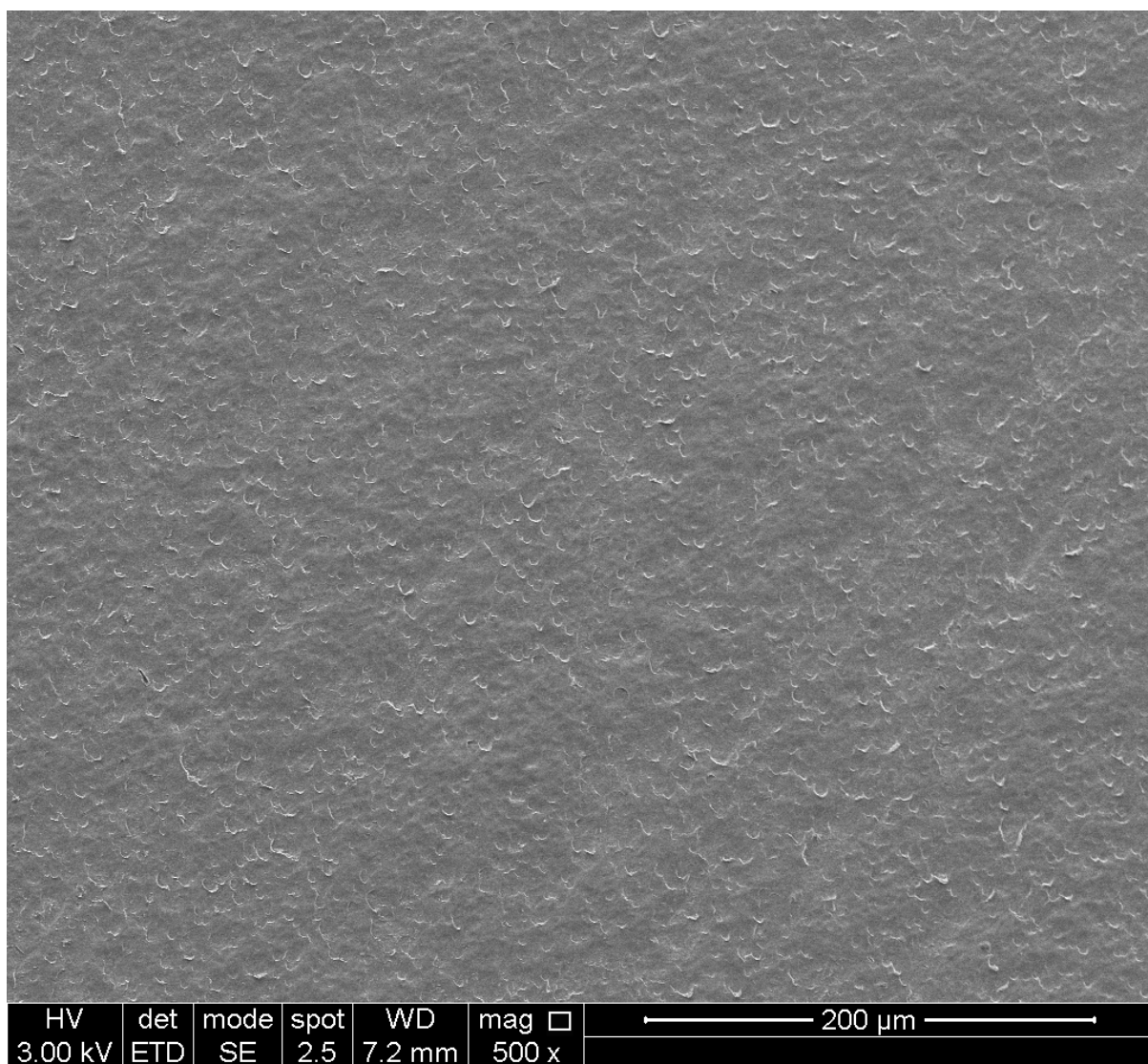


Figure 3a SEM of the CNF Nanopaper

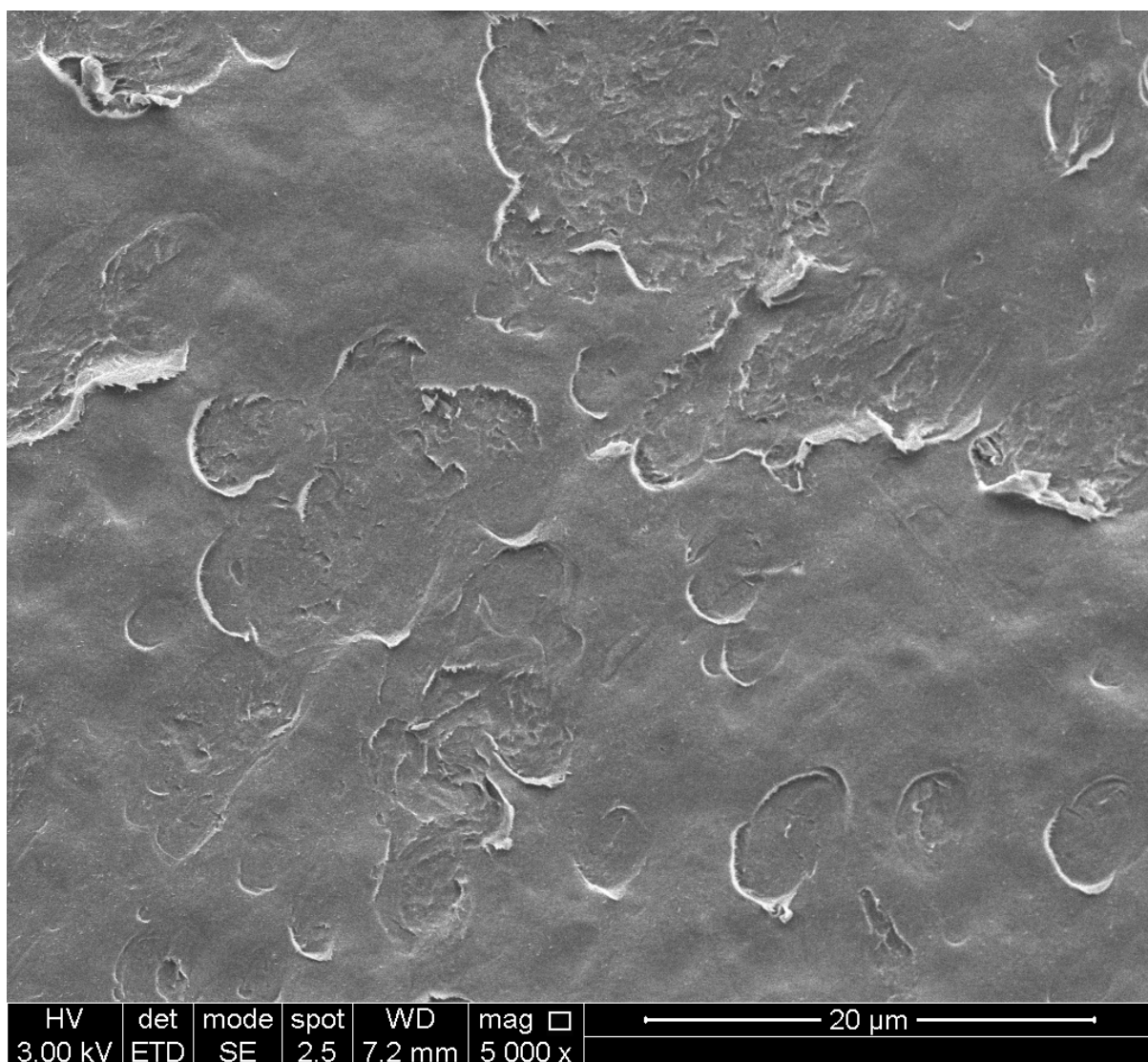


Figure 3b SEM of the CNF Nanopaper

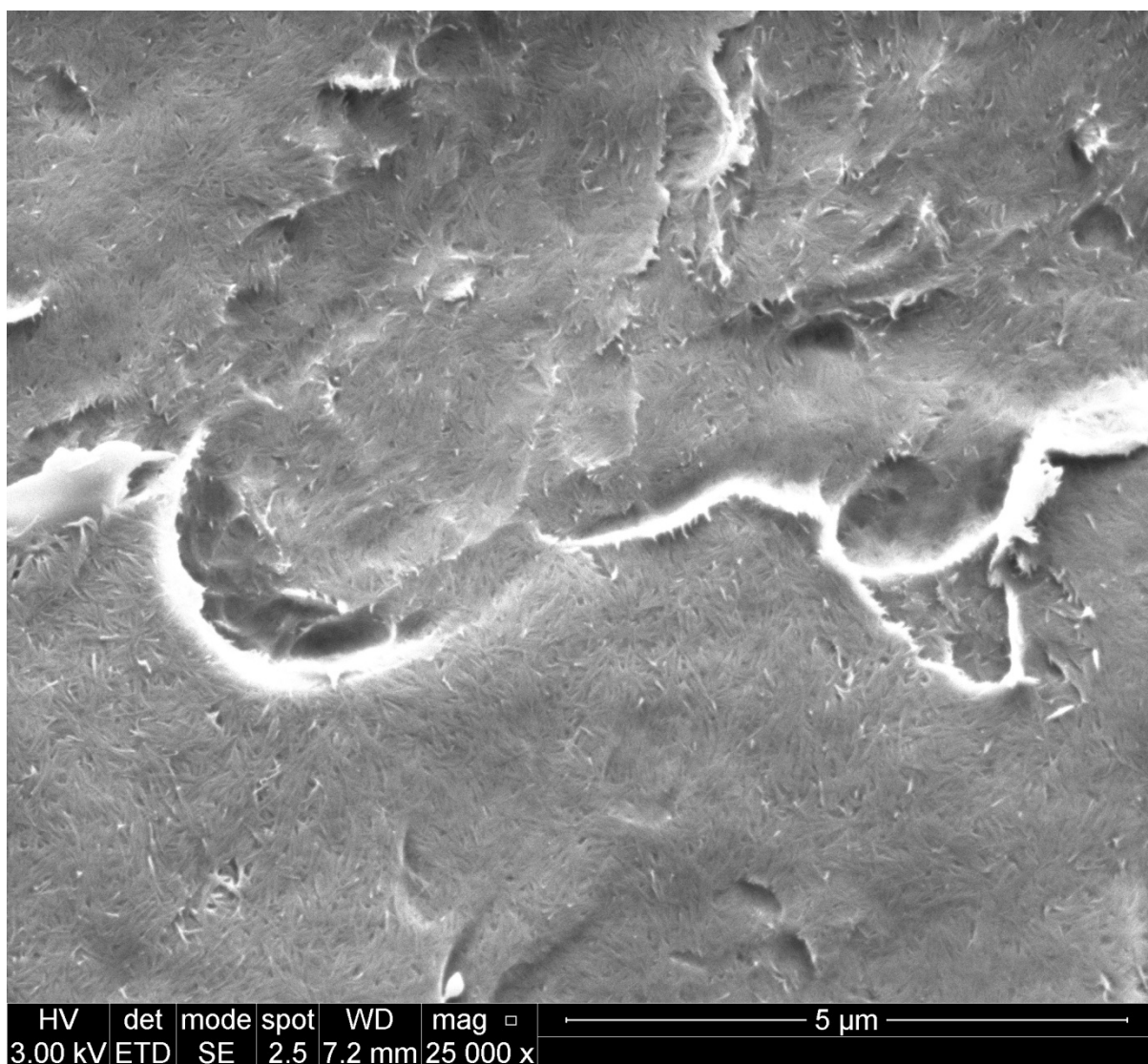


Figure 3c SEM of the CNF Nanopaper

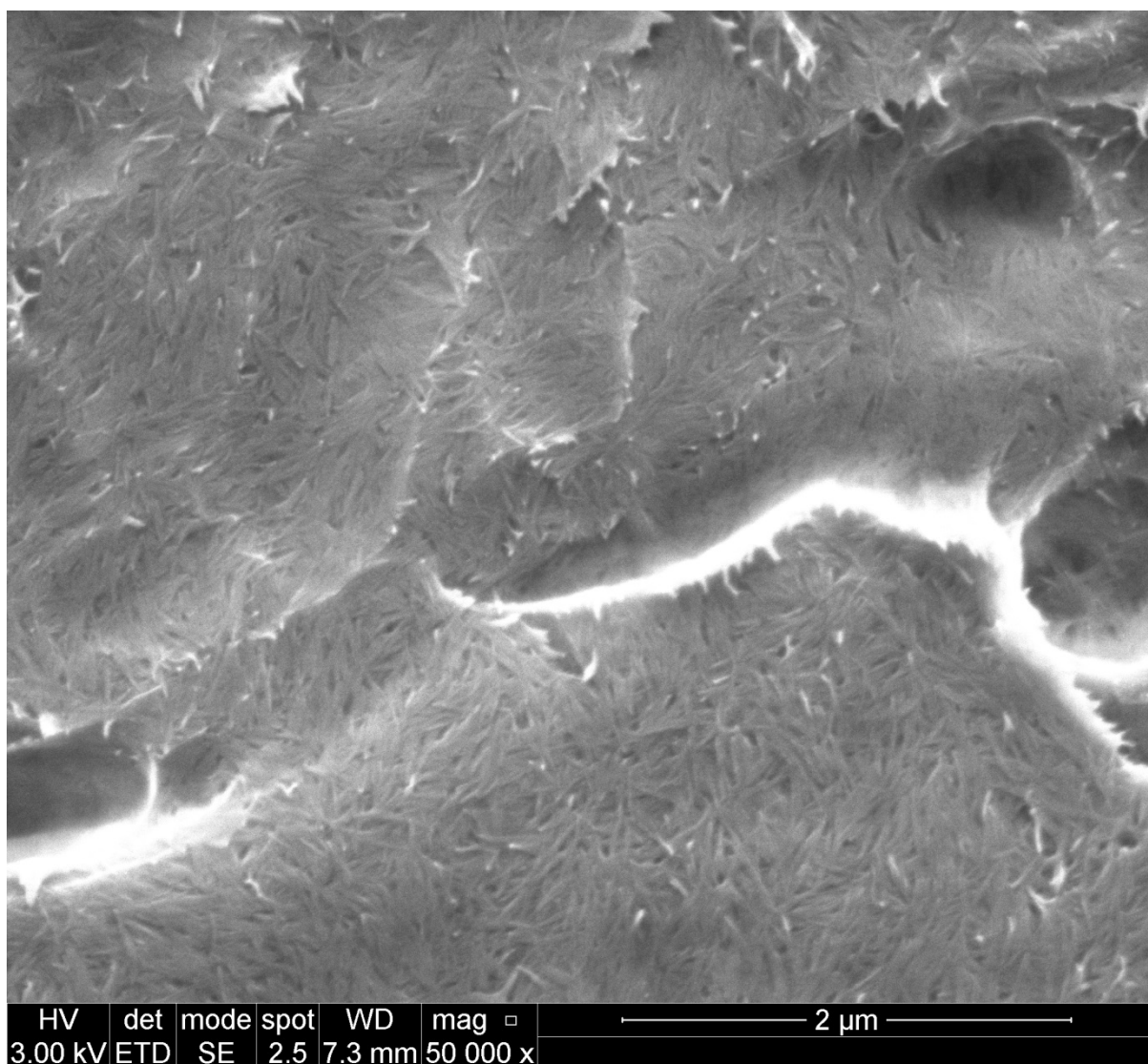


Figure 3d SEM of the CNF Nanopaper

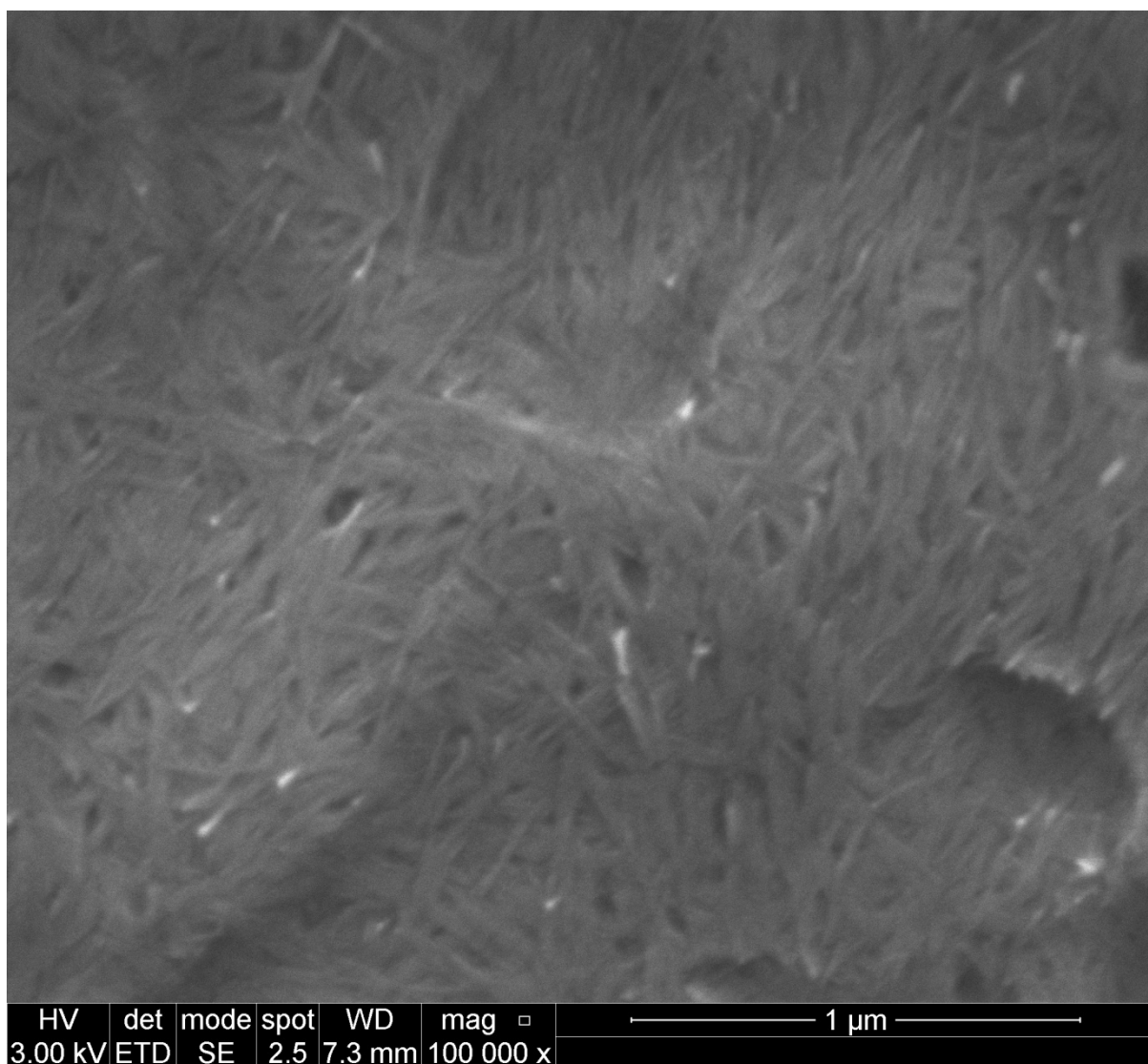


Figure 3e SEM of the CNF Nanopaper

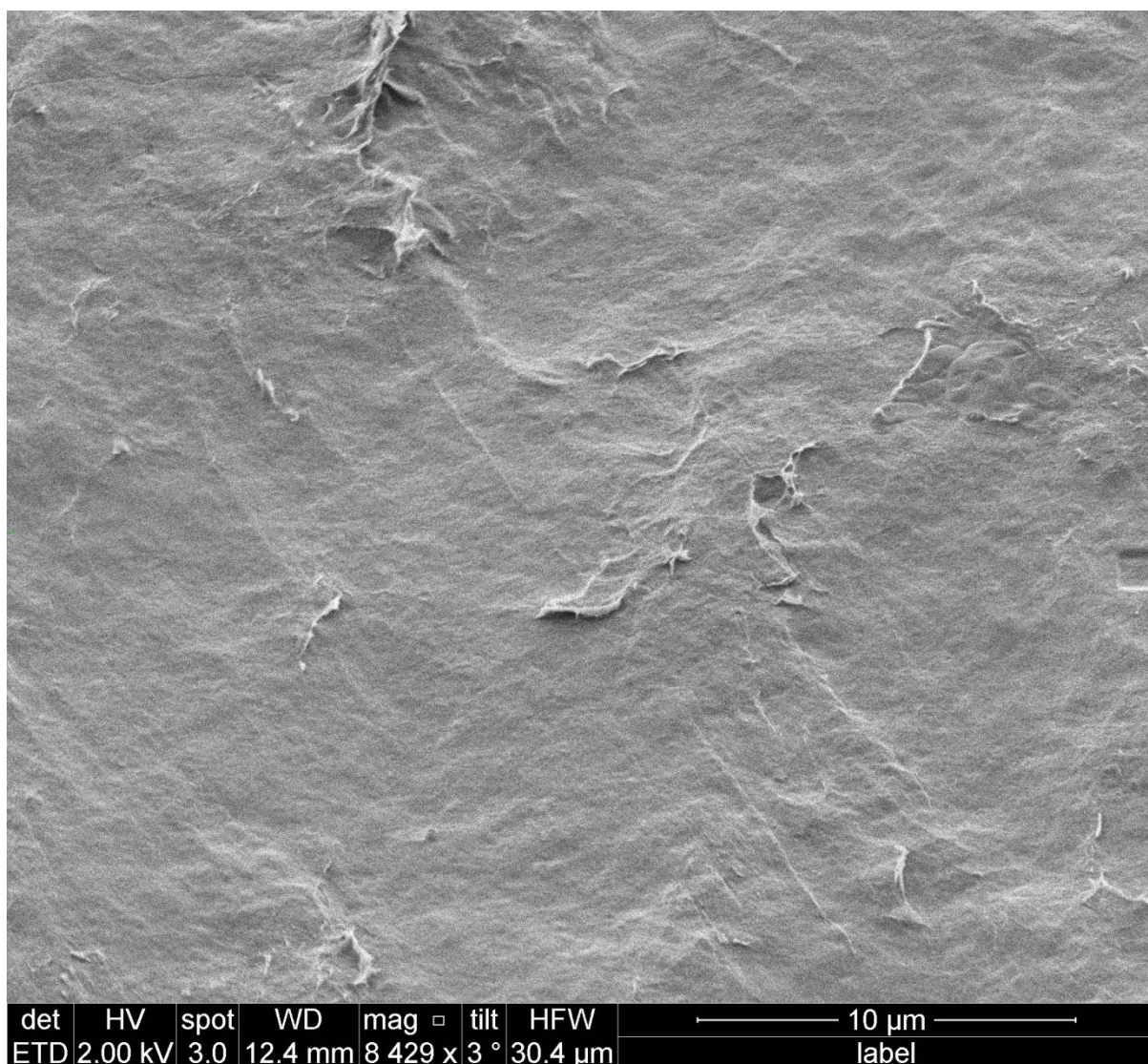
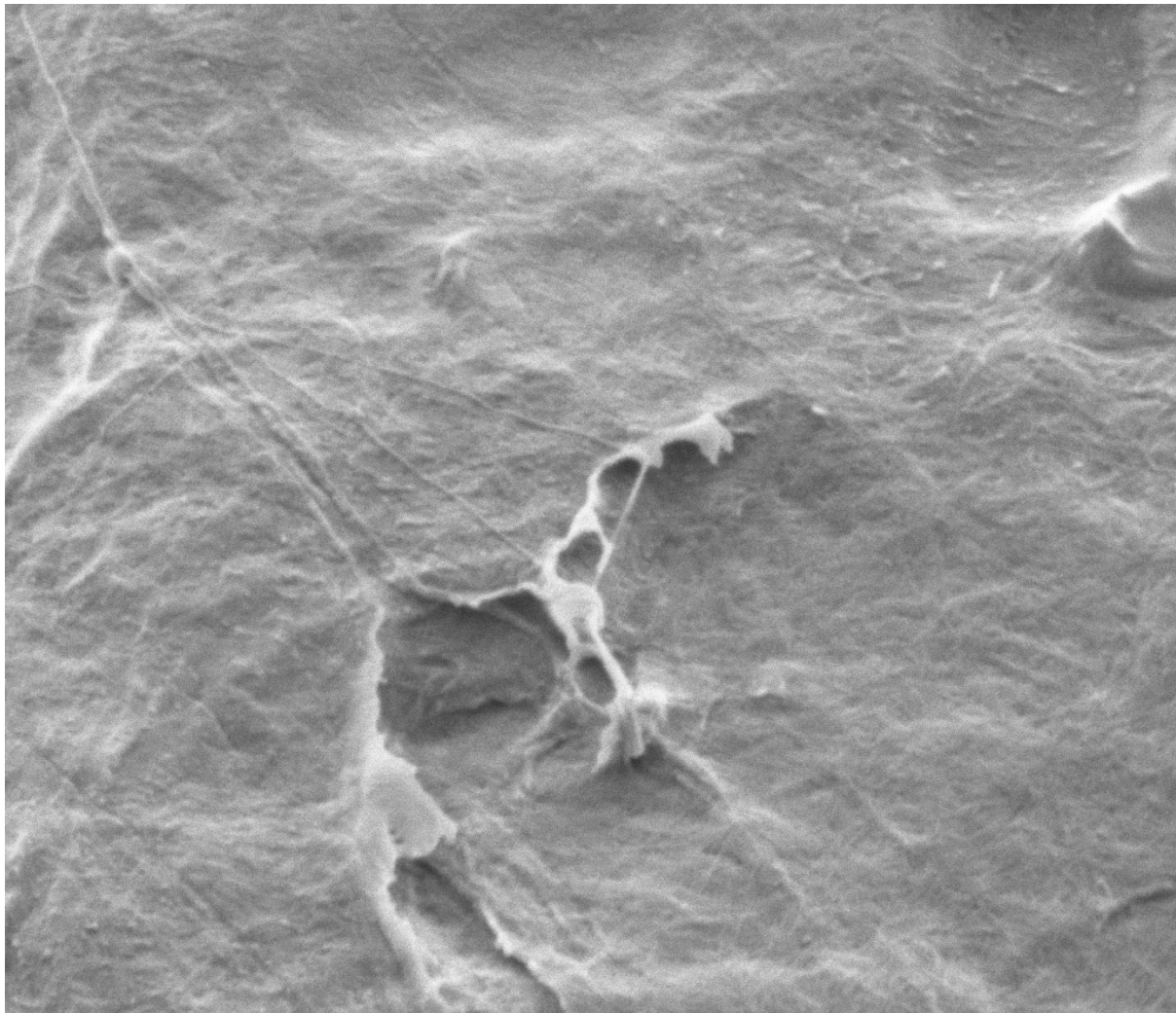


Figure 4a SEM of the CNF/Residue Nanopaper



det	HV	spot	WD	mag	□	tilt	HFW	2 μm
ETD	2.00 kV	3.0	12.4 mm	47 684 x		3 °	5.37 μm	label

Figure 4b SEM of the CNF/Residue Nanopaper

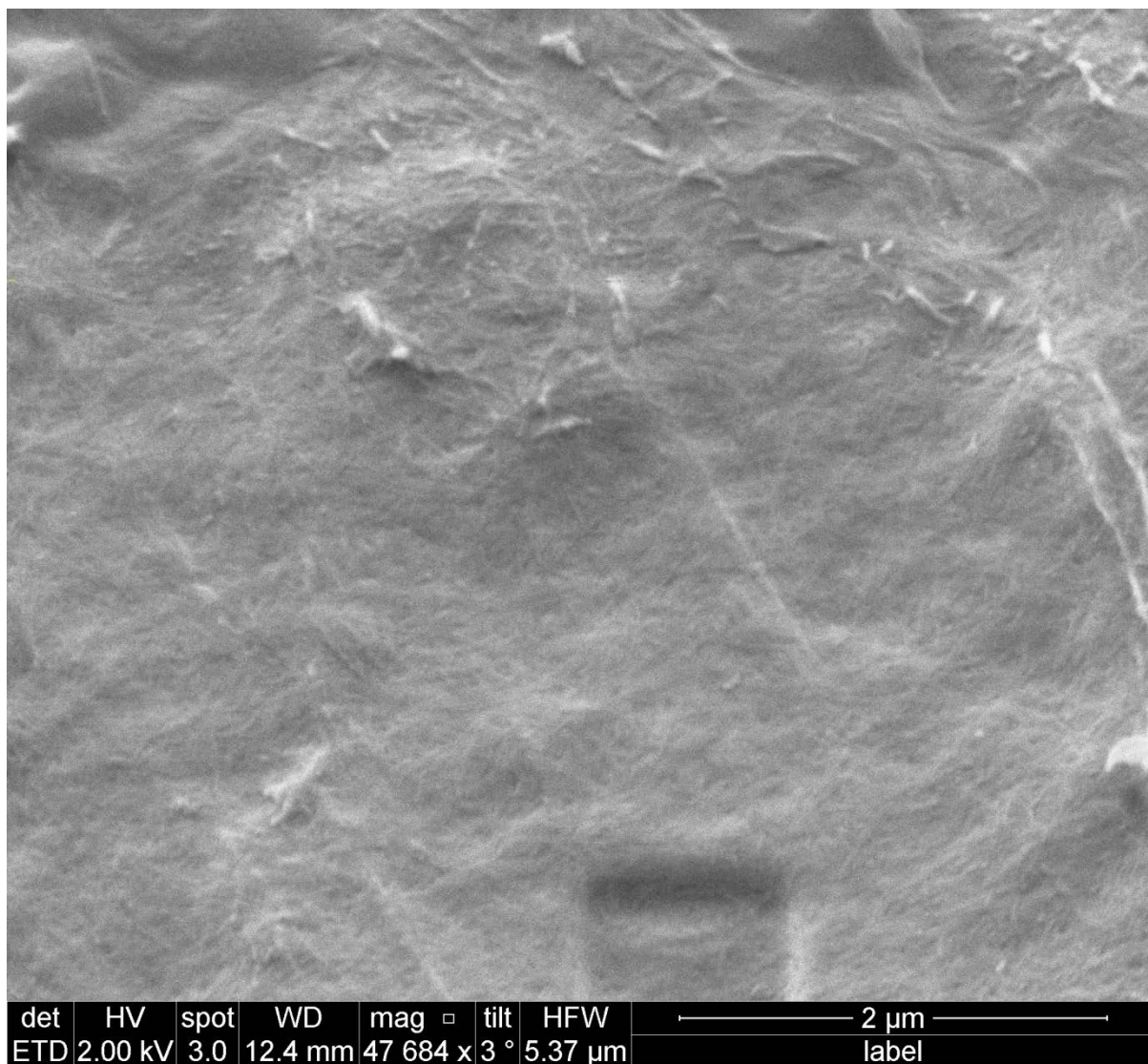


Figure 4c SEM of the CNF/Residue Nanopaper

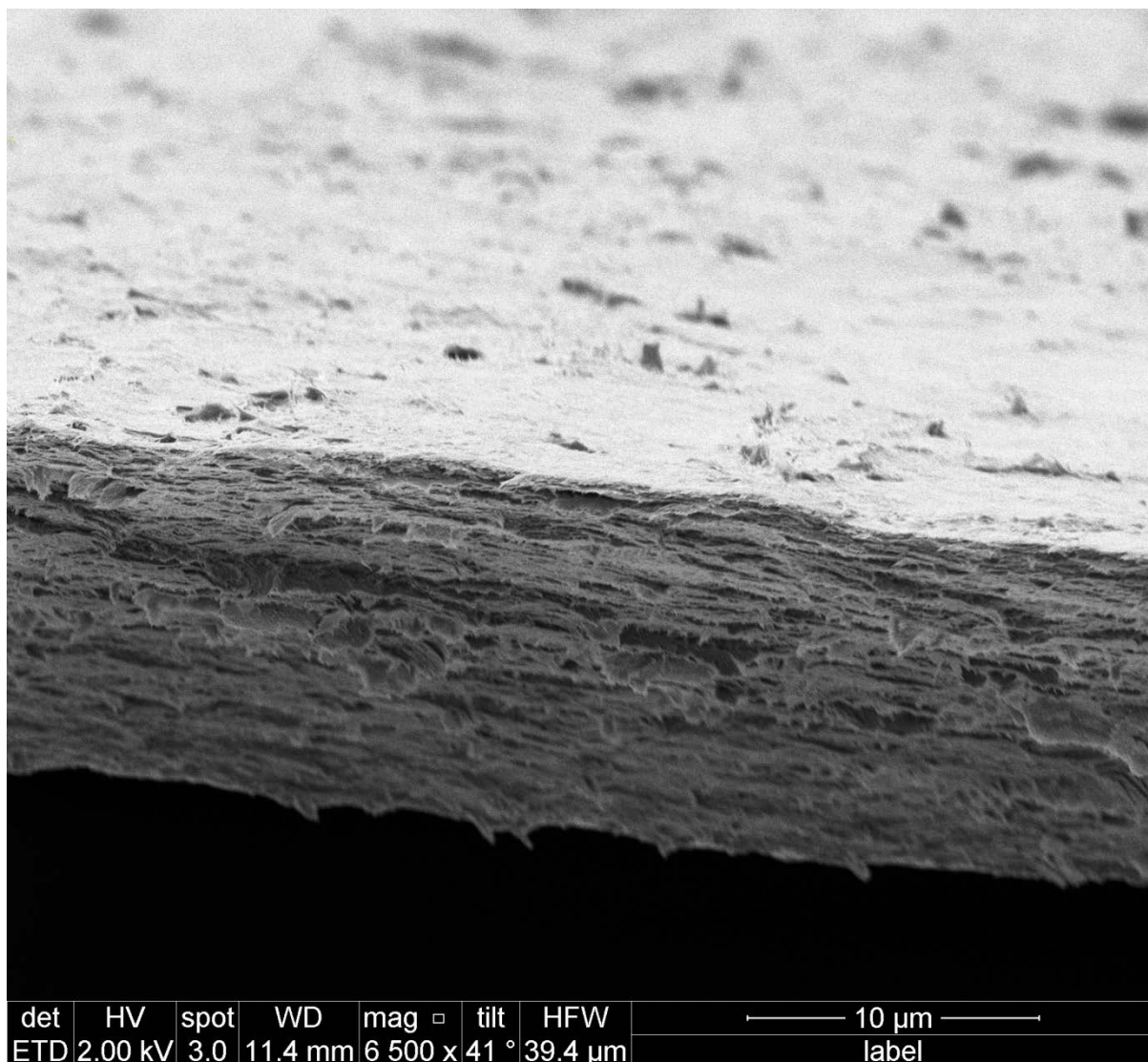


Figure 4d SEM of the CNF/Residue Nanopaper

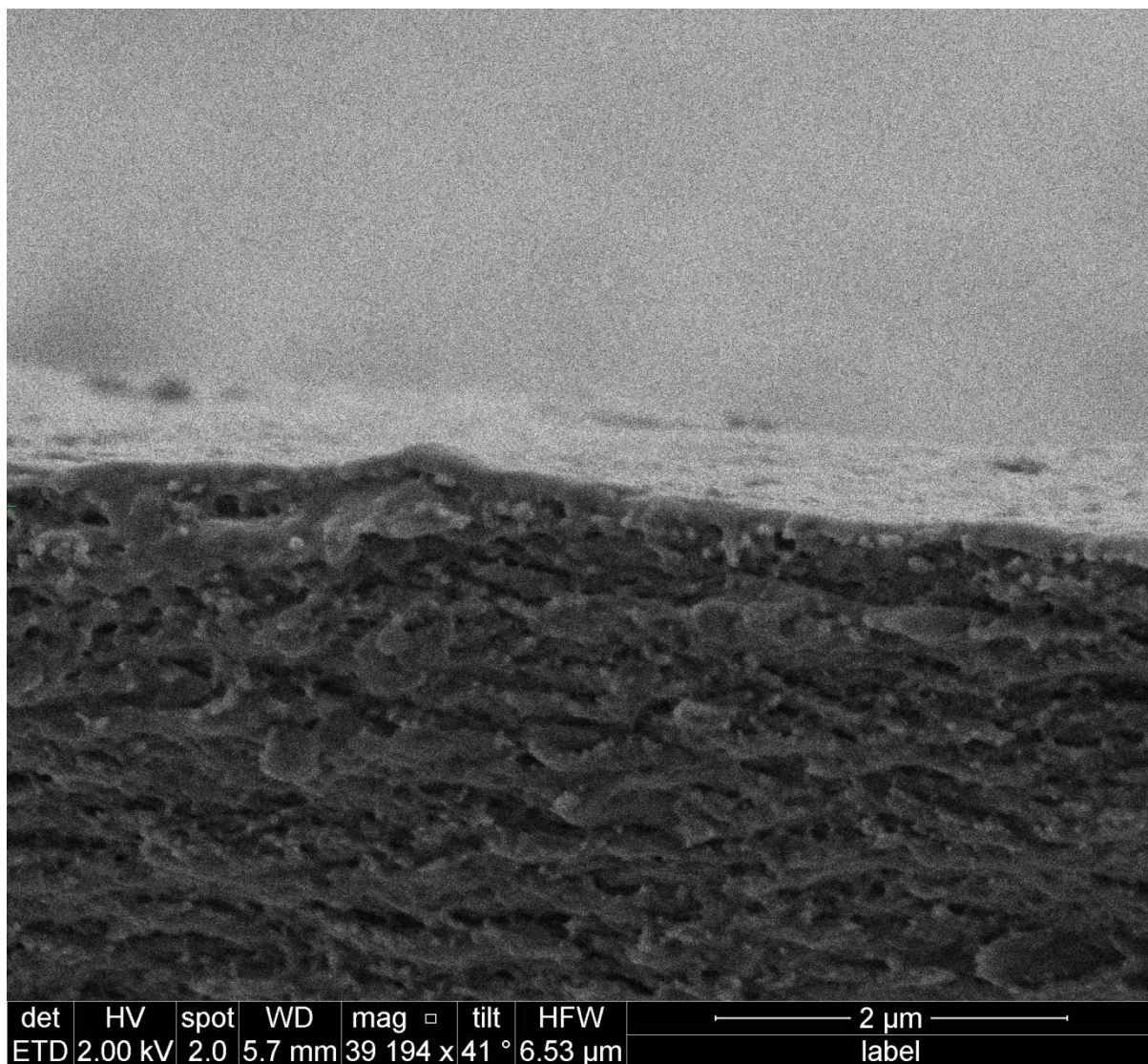


Figure 4e SEM of the CNF/Residue Nanopaper

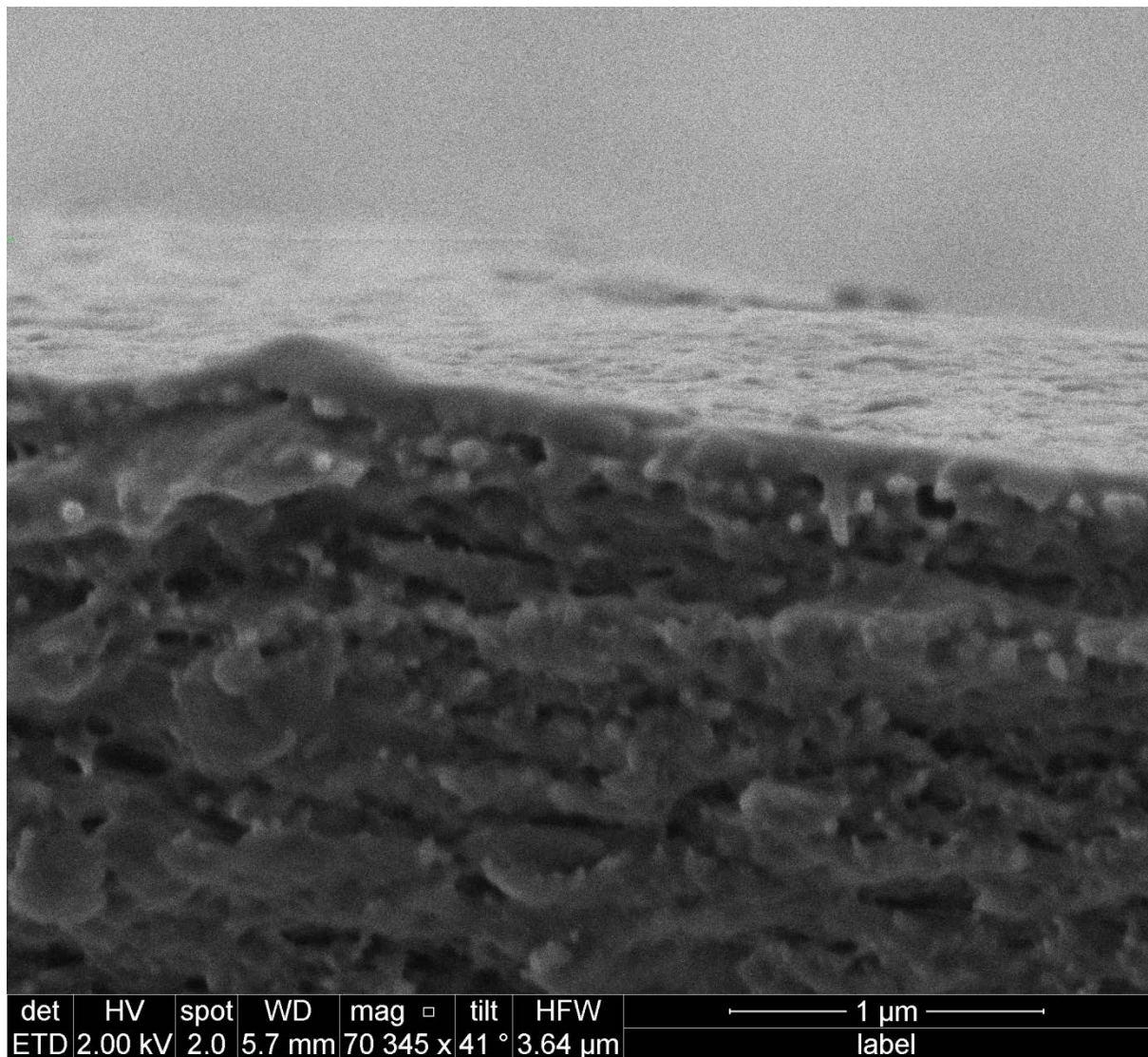


Figure 4f SEM of the CNF/Res at different magnifications

Table 1: Optical properties of the films determined by use of a hazemeter.

	CNF	CNF/Residue
Transparency	91,55% +/- 0,161	88,17% +/- 0,189
Haze	26,73% +/- 0,411	36,18% +/- 1,302
Clarity	69,77% +/- 1,157	44,58% +/- 1,771

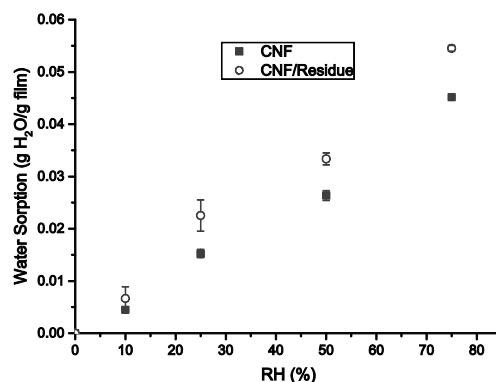


Figure 5 Water sorption of neat CNF and CNF/Residue film

Table 2 Mechanical properties of the CNF Films

	Stress (MPa)	Elongation at break (%)	E (GPa)
CNF	24,74 +/- 6,20	1,2 +/- 0,02	2,41 +/- 0,088
CNF/RES	42,59 +/- 4,46	2,65 +/- 0,29	1,810 +/- 0,044

The mechanical properties of the film were tested. Which in the case of the neat CNF film was very difficult to investigate due to the unevenness of the thin and brittle nanopaper. The neat CNF nanopaper was found to have poor mechanical properties, probably due to the unevenness of the film, while the nanopaper with the added residues showed improved mechanical properties, even though both films are very weak.

REFERENCES

- Fang, J., Sun, R., Tomkinson, J., 2000. Isolation and characterization of hemicelluloses and cellulose from rye straw by alkaline peroxide extraction. *Cellulose* 7, 87–107.
- Patil, R., Genco, J.M., Pendse, H., 2012. Cleavage of Acetyl Groups From Northeast Hardwood for Acetic Acid Production. *PEERS Conf.* 2177–2266.